REACTIONS IN THE SERIES OF SUBSTITUTED ISOINDOLO[1,2-*b*]-[3]-BENZAZEPIN-5-ONES*

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In the reaction of 10,11-methylenedioxy-3,4,12-trimethoxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3-]-benzazepin-5-one (*V*) with benzyl alcohol and sodium benzyloxide nucleophilic substitution of the 4-methoxy group for benzyloxy group takes place under formation of 4-benzyloxy-3,12-dimethoxy-10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*VI*). An analogous exchange converts compound *V* in the presence of corresponding alkoxides and alcohols to compounds *VII*—*X*. When reducing compound *V* with sodium dihydridobis(2-methoxyethoxy)aluminate the unstable base *XI* is formed which on reaction with trifluoroacetic acid gives the red trifluoroacetate *XII*. As a side product 3,12-dimethoxy-10,11-methylenedioxy-7,8-dihydroisoindolo[1,2-*b*][3]-benzazepin-5-one (*XIII*) is formed at the stage of the reduction of the nucleophilic substitution of the 4-methoxy group with the hydride anion. The reaction of compound *V* with various Grignard reagents always leads to the same product of phenolic character. Its structure *XIV* was confirmed by comparison of its mass spectra with those of the product of hydrogenolysis of compound *VI*. Compound *VI* and the product of benzylation of compound *XIV* were also identical.

Several years ago we described[1,2] how during thermal decomposition of methohydroxide of narceine imide (*I*) (*Z*)- and (*E*)-narceone imides (*II*) and *III*) are formed in addition to their products of cyclisation, *i.e.* 7-methyl-9,10-methylenedioxy-3,4,11-trimethoxy-6,7-dihydro-5*H*-isoindolo[1,2-*b*]isoquinol-5-one (*IV*) and 10,11-methylenedioxy-3,4,12-trimethoxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*V*). When investigating the structure of *IV* we observed[2] an easy exchange of one alkoxy group under the effect of the corresponding sodium alkoxide in alcohol. In this paper we describe some reactions of this type, which we observed in the isomeric compound *V*.

Heating of *V* with sodium benzyl oxide in benzyl alcohol and benzene gave a product which according to elemental analysis contains C_6H_4 more than the starting compound *V*. In its ^{1}H NMR spectrum the signals of a multiplet of 5 aromatic hydrogen atoms of the phenyl group appear at δ 7·15 to 7·75 ppm and of a singlet of 2 hydrogen atoms of the —O—CH_2—Ar group at δ 5·29 ppm, while the singlet at δ 4·05 ppm of the most shielded methoxy group in position 4, appearing only in

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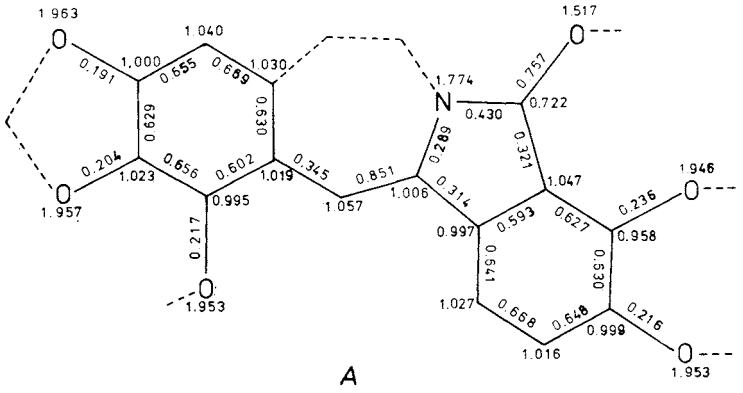
the ^{1}H NMR spectrum of the starting compound[1,2] *V*, is absent in the spectrum of the product. From this it follows that during the reaction nucleophilic substitution of the methoxy group for the benzyloxy group in position 4 takes place and the product can be assigned the structure *VI*. This formulation is in agreement with the quantum calculation of the π-electron densities and the π-order of bonds in the model system *A* (Scheme 1) which was carried out by a modified type[3] of LCI-SCF calculation using the Pariser, Parr, Pople method; for the details of calculation see ref.[3].



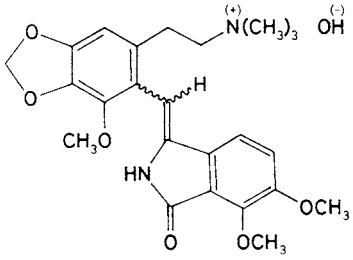
A

SCHEME 1

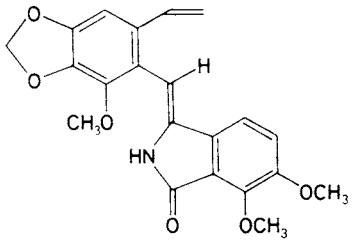
Analogously 4-ethoxy derivative *VII*, and 4-(2-(N,N-dimethylamino)ethoxy)-, 4-(2-(N-piperidino)ethoxy)- and 4-(2-(N-morpholino)ethoxy) derivatives *VIII*, *IX*, and *X* were prepared in reactions with corresponding alkoxides in alcohols.

We observed a further case of nucleophilic substitution in attempts at the reduction of compound *V* with sodium dihydridobis-(2-methoxyethoxy)aluminate. In this reaction a mixture of compounds is formed the main component of which is an unstable compound, *XI*, which is converted under the effect of trifluoroacetic acid to the red trifluoroacetate of the known[1] 10,11-methylenedioxy-3,4,12-trimethoxy--7,8-dihydroisoindolo[1,2-*b*][3]-benzazepinium (*XII*). As a by-product which we isolated from the mother liquors after trifluoroacetate *XII* we obtained a yellow neutral compound with m.p. 227–228°C with similar properties as the starting compound *V*, the elemental composition of which lacked CH_2O. From the comparison of its ^{1}H NMR spectrum with that of *V* it followed that it again does not contain the three-proton singlet of one methoxy group at δ 4·05 ppm, but it does contain a new singlet of the aromatic proton at δ 7·25 ppm which is *meta*-coupled with the signal of the doublet of the aromatic proton at δ 7·08 ppm, with the coupling constant J = 2·5 Hz. This situation can occur only if a nucleophilic substitution

of the methoxy group in position 4 with the hydride anion had taken place to a small extent under the effect of the reducing reagent. Therefore, the side product must have the structure *XIII*.



I



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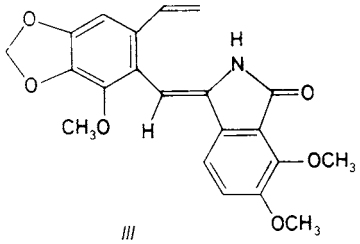
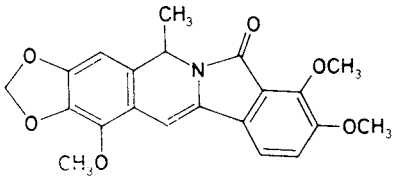
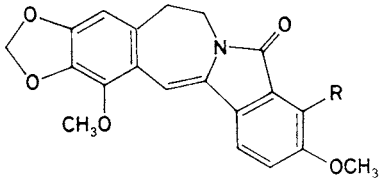


III



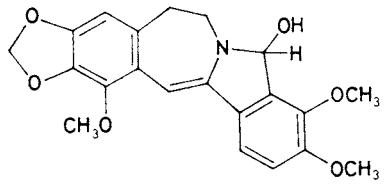
IV



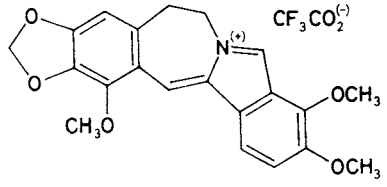
V, R = $-OCH_3$
VI, R = $-OCH_2-C_6H_5$
VII, R = $-OC_2H_5$
VIII, R = $-OCH_2CH_2-N(CH_3)_2$
IX, R = $-OCH_2CH_2-N$(morpholino)
X, R = $-OCH_2CH_2-N$(morpholino)
XIII, R = —H
XIV, R = —OH
XV, R = $-OCOCH_3$
XVI, R = $-OSO_2-C_6H_4-CH_3$

The relatively low reactivity of the carbonyl group of compound *V* also manifests itself in the reactions with the Grignard reagent. From the reaction mixture after the effect of methylmagnesium iodide, ethylmagnesium bromide or phenylmagnesium

bromide, we always isolated the same product of which we knew from the infrared spectrum that it contained a phenolic group, while its elemental composition was in agreement with the composition of the demethylation product. Acetylation with acetic anhydride gave the expected monoacetyl derivative *XV*, while reaction with *p*-toluenesulfonyl chloride in pyridine afforded mono-*p*-toluenesulfonate *XVI*. The position of the phenolic group could not be determined from the ^{1}H NMR spectrum unambiguously, but we assumed that again demethylation of the most protected methoxy group in position 4 had taken place. We confirmed this assumption by hydrogenolysing benzyloxy derivative *VI* to 4-hydroxy derivative with a guaranteed position of the phenolic function, which was identical in all respects with the products of demethylation of compound *V* with Grignard reagents. On benzylation of both phenolic products an identical benzyloxy derivative, *VI*, was formed. Hence, the demethylation product must be assigned the structure 3,12-dimethoxy-4-hydroxy--10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*XIV*).



XI



XII

EXPERIMENTAL

The melting points were determined on a Boetius microblock and they are not corrected. Samples for analysis were dried in a 10 Pa vacuum at room temperature for 8 h. The purity of the substances was checked by thin-layer chromatography on silica gel (Fertigplatten Merck Kieselgel 60 F_{254}) in benzene–ethanol 96 : 4 or toluene–acetone–ethanol–conc. ammonia 60 : 40 : 7·5 : 2·5. The spots were detected in UV light at 254 and 366 nm. The ultraviolet spectra (λ_{max} or λ_{min} nm (log ε)) were measured in methanol on a Varian DNS 90 instrument (Australia). The infrared spectra (ν, cm^{-1}) were measured on a Specord 75 IR Carl Zeiss (G.D.R.) instrument. The ^{1}H NMR spectra (δ, ppm) were measured on a Tesla BS 487 C instrument (Czechoslovakia). The mass spectra (m/z) were measured on an AEI MS 902 (70 eV) spectrometer.

4-Benzyloxy-3,12-dimethoxy-10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*]-[3]-benzazepin-5-one (*VI*)

A solution of 1·4 g of sodium in 70 ml of benzyl alcohol was added to a solution of 1·2 g of 10,11-methylenedioxy-3,4,12-trimethoxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*V*) in 300 ml of benzene and the mixture was refluxed for 4 h. After extraction with two 100 ml portions of a 10% aqueous sodium hydroxide solution and twice with 100 ml of water the organic phase was dried over anhydrous sodium sulfate and benzene evaporated. After distilling off benzyl alcohol in a vacuum (8 Pa) the residue was dissolved in benzene and filtered through

a column of neutral alumina (15 g; activity II—III). The residue after elimination of benzene afforded 0·92 g (64%) of compound *VI* (yellow needles), m.p. 129—131°C (benzene–light petroleum). For $C_{27}H_{23}NO_6$ (457·4) calculated: 70·88% C, 5·08% H, 3·06% N; found: 70·90% C, 5·34% H, 2·82% N. Ultraviolet spectrum: 226 (sh) (4·480), 271 (4·132), 318 (4·004), 384 (4·514). Infrared spectrum (chloroform): 1 688 (lactam), 1 605, 1 645 (aromatic vibrations and conjugated double bond), 1 495 (substitution on the aromatic ring). ^{1}H NMR spectrum (C^2HCl_3): 7·48 (d), 7·06 (d), J = 9·0 Hz (2 H, ABq; 2 *O*-H arom.); 7·15—7·75 (5 H, m; 5 H-arom.); 6·80 (1 H, s; olefinic); 6·38 (1 H, s; aromatic); 5·89 (2 H, s; OCH_2O); 5·29 (2 H, s; CCH_2Ar); 4·02 (3 H, s; OCH_3); about 4·00 (2 H, overlapped; CH_2); 3·81 (3 H, s; OCH_3); 2·94 (2 H, t; CH_2); totally 23 H.

4-Ethoxy-3,12-dimethoxy-10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*VII*)

A solution of compound *V* (1·14 g) in 120 ml benzene was added to a solution of 1·4 g sodium in 70 ml ethanol and the mixture was refluxed for 9 h. After shaking it twice with 100 ml of a 10% aqueous sodium hydroxide solution and twice with water the organic layer was dried over anhydrous sodium sulfate and the solvent evaporated. The residue was dissolved in benzene and filtered through a column of neutral alumina (25·0 g; act. II—III) and eluted with benzene. After evaporation of the solvent the residue was crystallized from benzene–light petroleum, to yield 1·02 g (86%) of product *VII*, m.p. 202—203°C. For $C_{22}H_{21}NO_6$ (395·4) calculated: 66·83% C, 5·35% H, 3·54% N; found: 66·82% C, 5·48% H, 3·51% N. Ultraviolet spectrum: 271 (4·108), 315 (3·977), 388 (4·498). Infrared spectrum (chloroform): 2 860 (OCH_3), 1 695 (carbonyl), 1 655, 1 616 (aromatic vibrations). ^{1}H NMR spectrum (C^2HCl_3): 7·48 (1 H, d, J = 8·0 Hz; aromatic); 7·07 (1 H, d, J = 8·0 Hz; aromatic); 6·79 (1 H, s; olefinic); 6·39 (1 H, s; aromatic); 5·90 (2 H, s; OCH_2O); 4·30 (2 H, q, J = 7·0 Hz; OCH_2CH_3); 4·02 (3 H, s; OCH_3); 3·90 (3 H, s; OCH_3); about 4·00 (2 H, m; CH_2); 2·98 (2 H, m; CH_2); 1·42 (3 H, t, J = 7·0 Hz; CH_2CH_3); totally 21 H.

3,12-Dimethoxy-4-(2-(N,N-dimethylamino)ethoxy)-10,11-methylenedioxy-7,8-dihydro-5H-isoindolo[1,2-*b*][3]-benzazepin-5-one (*VIII*)

A solution of 0·5 g of *V* in 50 ml benzene was added in portions to a partly cooled solution of 0·3 g of sodium in 3·1 g of 2-(N,N-dimethylamino)ethanol heated at 100—110°C. After stirring the mixture was refluxed for 5 h and cooled. After shaking with two 15 ml portions of 10% aqueous sodium hyroxide solution and two 15 ml portions of water the organic phase was dried over anhydrous sodium sulfate and the solvent was evaporated in a vacuum. The residue was dissolved in benzene and filtered through a column of 10 g of neutral alumina (act. II—III). After elution of the starting substance (benzene 200 ml) the product was eluted with chloroform (150 ml). Yield, 0·34 g (59%) of crude *VIII* which was crystallized twice from toluene with addition of a small amount of ethanol; m.p. 226—229°C (decomp.). Ultraviolet spectrum: λ_{max} 315 (3·94), 270 (4·05); λ_{min} 333 (3·90), 291 (3·76), 251 (3·89). Infrared spectrum (KBr pellet)*: 2 480, 2 600 (NH^+), 1 680 (C=O), 1 600, 1 630 (aromatic vibrations), 1 270 (C—O—Ar), 1 060 (C—O—Alkyl). ^{1}H NMR spectrum ($C^2H_3SOC^2H_3$, 80°C): 7·60 (1 H, d, J = 8·5 Hz; aromatic); 7·30 (1 H, d, J = 8·5 Hz; aromatic); 6·78 (1 H, s; olefinic); 6·50 (1 H, s; aromatic); 5·92 (2 H, s; OCH_2O); 4·45 (2 H, t; OCH_2); 3·98 (3 H, s; OCH_3); 3·88 (3 H, s; OCH_3); about 3·85 (2 H, bt; CH_2); 3·48 (2 H, t; OCH_2CH_2N=); 2·95 (2 H, bt; $ArCH_2CH_2$); 2·92 (6 H, s; 2 CH_3); totally 26 H.

* (measured as hydrochloride)

3,12-Dimethoxy-10,11-methylenedioxy-4-(2-(N-piperidino)ethoxy)-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*IX*)

A solution of *V* (500 mg) in 50 ml of benzene was added to a partly cooled solution of 0·3 g of sodium dissolved in 5·8 g of 2-(N-piperidino)ethanol at 100—120°C and after stirring the mixture was refluxed for 5 h. After coolling the solution was shaken twice with 15 ml of a 10% aqueous solution of sodium hydroxide and washed with two 15 ml portions of water and the organic phase was dried over anhydrous sodium sulfate, filtered and the solvent evaporated in a vacuum. The residue was dissolved in benzene and filtered through a column of neutral alumina (12 g; act. II—III). The starting compound (162 mg) was eluted with benzene (450 ml) and the product *IX* with chloroform (250 ml). The residue (373 mg) was crystallized from acetone–light petroleum. Yield, 252 mg of *IX* (40%),m.p. 238—242°C. Ultraviolet spectrum: λ_{max} 385 (4·49), 315 (4·00), 270 (4·12); λ_{min} 334 (3·95), 292 (3·84), 253 (3·96). Infrared spectrum (KBr pellet): 2 480, 2 600 (NH^+)*, 1 690 (C=O), 1 580, 1 620 (aromatic vibrations), 1 260 (C—O—Ar), 1 080 (C—O—Alkyl). ^{1}H NMR spectrum ($C^2H_3SOC^2H_3$, 120°C): 7·60 (1 H, d, J = 9·0 Hz; aromatic); 7·30 (1 H, d, J = 9·0 Hz; aromatic); 6·80 (1 H, s; olefinic); 6·50 (1 H, s; aromatic); 5·97 (2 H, s; OCH_2O); 4·58 (2 H, t; $OCH_2CH_2N<$); 4·00 (3 H, s; OCH_3); 3·90 (3H, s; OCH_3); about 3·95 (2 H, m; CH_2); 3·40, 3·00 (6 H, m; $CH_2N\langle_{CH_2}^{CH_2}\rangle_2$); 1·80 (6 H, m; 3 CH_2 in piperidine); totally 30 H.

3,12-Dimethoxy-4-(2-(N-morpholino)ethoxy)-10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*X*)

A solution of compound *V* (0·5 g) in 50 ml benzene was added to a partly cooled solution of 0·3 g of sodium in 5·0 g of 2-(N-morpholino)ethanol, prepared at 120°C. After stirring the mixture was refluxed for 5 h, cooled and extracted with two 15 ml portions of a 10% sodium hydroxide solution in water and twice with 15 ml of water. The organic phase was dried over anhydrous sodium sulfate and benzene evaporated in a vacuum. The residue was dissolved in benzene and filtered through a column of silica gel (15 g). Elution with benzene (3 500 ml) gave the starting compound *V* (0·06 g) and elution with chloroform (600 ml) the product (0·40 g, 64%). Compound *X* was crystallized from acetone, m.p. 157—161°C. Ultraviolet spectrum: λ_{max} 315 (3·98), 269 (4·10); λ_{min} 332 (3·95), 289 (3·80), 253 (3·96). Infrared spectrum (KBr pellet): 1 680 (C=O), 1 600, 1 640 (aromatic vibrations), 1 260 (C—O—Ar), 1 060 (C—O—Alkyl). ^{1}H NMR spectrum ($C^2H_3SOC^2H_3$): 7·56 (1 H, d, J = 8·5 Hz; aromatic); 7·21 (1 H, d, J = 8·5 Hz; aromatic); 6·70 (1 H, s; olefinic); 6·50 (1 H, s; aromatic); 5·95 (2 H, s; OCH_2O); 4·21 (2 H, t, J = 6·0 Hz; OCH_2); 3·98 (3 H, s; OCH_3); 3·85 (3 H, s; OCH_3); 3·85 (2 H, m; $CH_2N<$); 3·52 (4 H, m; CH_2OCH_2); 2·95 (2 H, m; $ArCH_2CH_2$); 2·80 (2 H, t, J = 6·0 Hz $OCH_2CH_2N\langle$); 2·55 (4 H, m; $CH_2N\langle_{CH_2}^{CH_2}\rangle_2$); totally 28 H.

4-Hydroxy-3,12-dimethoxy-10,11-methylenedioxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*XIV*)

A solution of methylmagnesium iodide (from 0·28 g of magnesium and 1·6 g of methyl iodide in 6 ml of ether) was added dropwise and under stirring to a solution of 1·16 g of *V* in 180 ml of benzene and the mixture was refluxed for 5 h. Decomposition was carried out by addition of 5 ml of a saturated ammonium chloride solution and 5 ml of water, the separated solid phase

* (measured as hydrochloride)

was filtered off under suction, washed with benzene and water, the benzene phase was washed three times with 20 ml of a 5% aqueous sodium hydroxide solution, once with 20 ml of water and dried over anhydrous sodium sulfate. After filtration and evaporation of benzene 76 mg of compound *V* (6·8%) were regenerated. On crystallization of the solid fraction from dilute acetic acid 0·66 g (59%) of *XIV* were obtained, m.p. 211–215°C. For $C_{20}H_{17}NO_6$ (367·3) calculated: 65·39% C, 4·66% H, 3·82% N; found: 65·20% C, 4·71% H, 4·03% N. Ultraviolet spectrum: λ_{max} 385 (4·44), 311 (3·89), 268 (3·07); λ_{min} 333 (3·73), 288 (3·76), 252 (3·88). Infrared spectrum (KBr pellet): 3 350 (OH), 2 830 (OCH_3), 1 680 (amide), 1 640, 1 600 (aromatic vibrations). The same product *XIV* was also obtained on reaction of compound V with ethylmagnesium bromide (32%) and phenylmagnesium bromide (29%).

Acetate *XV*

This was prepared by refluxing 200 mg of compound *XIV* with 2 ml of acetic anhydride for 2 h and crystallization of the crude product *XV* from a large volume of benzene. Yield, 146 mg (65·5%), m.p. 255–260°C (decomp.). For $C_{22}H_{19}NO_7$ (409·3) calculated: 64·54% C, 4·68% H, 3·42% N; found: 64·43% C, 4·84% H, 3·56% N. Ultraviolet spectrum: λ_{max} 280 (4·42), 316 (3·97), 268 (4·05); λ_{min} 289 (3·78), 250 (3·88). Infrared spectrum (KBr pellet): 2 830 (OCH_3), 1 680 (amide in a five-membered ring), 1 780 (aryl acetate), 1 620, 1 600 (aromatic vibrations).

p-Toluenesulfonate *XVI*

This was prepared on reaction of 0·37 g of compound *XV* with an excess of *p*-toluenesulfonyl chloride in pyridine at room temperature, by standing for 48 h. After the conventional work-up 0·47 g of pure product *XVI* (90%) were obtained, which was crystallized from acetone, m.p. 232–235°C. For $C_{27}H_{23}NO_8S$ (521·5) calculated: 62·18% C, 4·44% H, 2·69% N, 6·15% S; found: 62·03% C, 4·42% H, 3·03% N, 6·43% S.

Hydrogenation of 4-benzyloxy derivative *VI*

0·5 g of 10% palladium on charcoal was added to a solution of 0·917 g of 4-benzyloxy derivative *VI* in 50 ml of acetic acid and the mixture was shaken under hydrogen at room temperature and atmospheric pressure until the consumption of hydrogen ceased. The separated product was brought into solution by heating and the catalyst was filtered off. On cooling of the filtrate 390 mg of *XIV* (53%) separated, with m.p. 215–217°C. Mass spectrum: 367 (the base and molecular peak, $C_{20}H_{17}NO_6$), 352, 324, 279 and 183. A similar fragmentation and the same peaks are observed in samples of *XIV* which were obtained by reaction of *V* with methylmagnesium iodide, ethylmagnesium bromide and phenylmagnesium bromide.

Benzylation of 4-hydroxy derivative *XIV*

A solution of 16·3 mg of 18-crown-6-polyether in 10 ml of benzene was added to 42·6 mg of a 75% dispersion of sodium hydride in paraffin oil, overlayered with 7 ml of benzene over 10 min. The addition was carried out under nitrogen and stirring and the mixture was refluxed under stirring for 15 min. Then a solution of 180 mg of benzyl bromide in 10 ml of benzene was added dropwise over 15 min and the mixture was refluxed for 5 h when another 180 mg of benzyl bromide in 10 ml of benzene and 10 ml of hexamethylphosphoric triamide (HMPT) were added and the refluxing continued for another 24 h. After decomposition with 10 ml of water the organic layer was extracted three times with 30 ml of water, three times with 30 ml of a 10% aqueous sodium hydroxide solution and twice with 50 ml of water, and the benzene layer was

dried over anhydrous sodium sulfate and evaporated. The residue was dissolved in benzene and filtered through a layer of 3 g of neutral alumina (act. II—III, benzene). The residue of the benzene fraction was crystallized from benzene–light petroleum to yield 40 mg (10%) of chromatographically pure 4-benzyloxy derivative *VI*, m.p. 130—132°C, which was identical in all parameters and spectra with the product obtained by nucleophilic substitution of compound V with sodium benzyl oxide and benzyl alcohol.

Reduction of compound *V* with sodium dihydridobis-(2-methoxyethoxy)aluminate

A 50% solution of Synhydride in toluene (5 ml) diluted with 15 ml of benzene was added dropwise to a solution of 1·55 g of compound *V* under exclusion of humidity. The addition lasted 15 min and the mixture was then heated at 60°C for 4 h. Decomposition was carried out by gradual addition of 5 ml of water, 5 ml of 15% aqueous sodium hydride solution and 15 ml of water, the benzene layer was separated, washed with water and dried over anhydrous sodium sulfate; 20 ml of a 2 mol l^{-1} solution of trifluoroacetic acid in benzene were then added to a benzene solution of *XI* and the separated crystals were filtered off under suction and dried in a vacuum desiccator over phosphorus pentoxide and potassium hydroxide until the weight no longer changed. Yield, 0·6 g (31%) of green crystals, m.p. 140—143°C, soluble in methanol or chloroform to a red-coloured solution. Ultraviolet spectrum: λ_{max} 266 (4·213), 390 (4·128), 626 (4·564). ^{1}H NMR spectrum (trifluoroacetic acid): 8·95 (1 H, s; $\overset{(+)}{>}N{=}CH{-}$); 8·59 (1 H, s; $ArCH{=}C\begin{matrix}\diagup N^{(+)} \\ \diagdown Ar\end{matrix}$); 7·88 (1 H, d, J = 9·0 Hz; aromatic); 7·62 (1 H, d, J = 9·0 Hz; aromatic); 6·64 (1 H, s; aromatic); 6·20 (2 H, s: OCH_2O); 4·70 (2 H, t; $\overset{(+)}{CH_2}N$); 4·29 (3 H, s; OCH_3); 4·06 (3 H, s; OCH_3); 3·35 (2 H, t; $ArCH_2CH_2$); totally 20 H. The compound was assigned the structure of 10,11-methylenedioxy-3,4,12-trimethoxy-7,8-dihydroisoindolo[1,2-*b*][3]benzazepinium trifluoroacetate (*XII*).

The benzene, blue mother liquor was evaporated in a vacuum, the residue was dissolved in acetone and filtered. The filtrate was poured slowly under stirring into 350 ml of light petroleum and the crystalline substance precipitated was filtered off. The light petroleum solution was evaporated in a vacuum and the green-yellow residue was dissolved in benzene and filtered through a column (20 g) of neutral alumina (act. II—III). Elution with benzene and evaporation in a vacuum gave a residue from which crystallization from benzene afforded 0·31 g (22%) of 10,11-methylenedioxy-3,12-dimethoxy-7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]-benzazepin-5-one (*XIII*), m.p. 227—228°C. For $C_{20}H_{17}NO_5$ (351·3) calculated: 68·37% C, 4·88% H, 3·99% N; found: 67·99% C, 5·40% H, 3·77% N. Ultraviolet spectrum: λ_{max} 234 (4·360), 240 (4·318), 272 (4·108), 311 (4·046), 370 (4·427). Infrared spectrum (chloroform): 1 688 (lactam), 1 610, 1 618, 1 645 (aromatic vibrations and conjugated double bond), 1 495 (substitutions on aromatic ring). ^{1}H NMR spectrum (C^2HCl_3): 7·68 (1 H, d, J = 9·0 Hz; aromatic); 7·25 (1 H, mcs, J = 2·5 Hz; aromatic); 7·08 (1 H, mcd, J = 9·0 Hz, 2·5 Hz; aromatic); 6·79 (1 H, s; $ArCH{=}C\langle$); 6·35 (1 H, s; aromatic); 5·88 (2 H, s; OCH_2O); 4·01 (3 H, s; OCH_3); about 4·00 (2 H, t: CH_2N^{CO}); ·81 (3 H, s; OCH_3); 2·92 (2 H, t, J = 4·0 Hz; $ArCH_2$); totally 17 H.

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